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BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte MICHEL STREBELLE and JEAN-PIERRE CATINAT

Appeal[1] 2009-008373
Application 10/534,299
Technology Center 1600

Decided: April 27, 2010

Before ERIC GRIMES, JEFFREY N. FREDMAN, and
STEPHEN WALSH, *Administrative Patent Judges*.

FREDMAN, *Administrative Patent Judge*.

DECISION ON APPEAL

This is an appeal under 35 U.S.C. § 134 involving claims to methods of manufacturing 1,2-epoxy-3-chloropropane. We have jurisdiction under 35 U.S.C. § 6(b). We affirm-in-part.

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[1] Oral Hearing held on April 22, 2010.

Statement of the Case

Background

"It is known to manufacture 1,2-epoxy-3-chloropropane (or epichlorohydrin) by epoxidation of allyl chloride by means of hydrogen peroxide in the presence of methanol as solvent and in the presence of a catalyst" (Spec. 1, ll. 4-6). According to the Specification, the "process exhibits the disadvantage that the catalyst rapidly deactivates, sometimes after a few hours of operation" (Spec. 1, ll. 8-9).

The Specification teaches that "the use of purified allyl chloride makes it possible to increase the duration of use of the catalyst (and thus to reduce the frequency with which the catalyst has to be removed from the epoxidation medium in order to be replaced) while retaining a high activity and a high selectivity" (Spec. 1, ll. 25-29).

The Claims

Claims 1-16 are on appeal. Claims 1 and 3 are representative and read as follows:

> 1. A process for the manufacture of 1,2-epoxy-3-chloropropane, comprising reacting allyl chloride and hydrogen peroxide in the presence of a zeolite catalyst and in the optional presence of at least one solvent, wherein the allyl chloride comprises less than 2000 ppm by weight of 1,5-hexadiene.
>
> 3. The process according to Claim 2, wherein the allyl chloride comprises less than 200 ppm by weight of 1,5-hexadiene.

The prior art

The Examiner relies on the following prior art references to show unpatentability:

Strebelle et al.	US 6,288,248 B1	Sep. 11, 2001
Fukuda[2] (as translated)	JP 4-327582	Nov. 17, 1992

The issue

The Examiner rejected claims 1-16 under 35 U.S.C. § 103(a) as obvious over Strebelle and Fukuda (Final Rej. 2-4). The Examiner finds that "Strebelle et al. disclose all of the claimed limitations except the use of an allyl chloride comprising less than 2000 ppm by weight of 1,5-hexadiene" (Final Rej. 4). The Examiner finds that Fukuda teaches that it is "desirable to utilize an allyl chloride comprising a 1,5-hexadiene content below 0.1 weight % (1000 ppm)" (Final Rej. 4). The Examiner concludes it obvious "to utilize an allyl chloride having a 1,5-hexadiene content below 0.1 weight % (1000 ppm) . . . in the process of Strebelle et al. because it would allow the artisan to prepare the epichlorohydrin of Strebelle et al. without formation of the unwanted by-product, 1,2-epoxy-5-hexene" (Final Rej. 4).

Appellants argue that "in a three component reaction - reactant, oxidizing agent and catalyst - Strebelle and Takehisa differ significantly in their choice of two of the three. These are not analogous reactions. While the product of the two reactions is the same, the mechanisms of the reactions are necessarily different" (App. Br. 5). Appellants argue that "[t]his noncombinability of Strebelle and Takehisa is further evident in view of the

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[2] The Examiner sometimes refers to this reference as Takehisa (*see, e.g.*, Final Rej. 3). We will refer to the reference as Fukuda.

1998 article by Sheldon, originally submitted to the Examiner on October 29, 2008, wherein Takehisa's catalysts are described as ineffective with Strebelle's peroxide" (Reply Br. 5).

Appellants argue that "unexpected improvement in catalyst life as provided by the present invention has been amply demonstrated in the present specification" (App. Br. 6). Appellants argue that "production using normal allyl chloride is exhausted at 27 hours, while allyl chloride according to the present invention remains active at 147 hours of reaction time" (App. Br. 7).

In view of these conflicting positions, we frame the obviousness issues before us as follows:

(1) Does the evidence support the Examiner's conclusion that the cited references would have suggested the method of claim 1?

and, if so,

(2) Have Appellants presented evidence of unexpected results that outweighs the evidence supporting a conclusion of obviousness?

Findings of Fact (FF)

1. Strebelle teaches:

> [a] process for manufacturing an epichlorohydrin-based product containing at least 99.9% by weight of epichlorohydrin and a total amount of chloro impurities of less than or equal to 150 ppm by weight, in which (a) allyl chloride is reacted with a peroxide compound in the presence of water, a catalyst and a diluent in at least one reactor, and (b) the reaction mixture leaving step (a) is subjected to a treatment to separate out the epichlorohydrin.

(Strebelle, col. 1, l. 60 to col. 2, l. 2).

2. Strebelle teaches that the "catalysts which can be used in step (a) of the process according to the invention are preferably titanium silicalite type catalysts. These are crystalline synthetic materials similar in structure to zeolites, comprising silicon oxide and titanium oxide" (Strebelle, col. 2, ll. 3-7).

3. Strebelle teaches that the "poor miscibility of the reagents, the allyl chloride and the aqueous solution of peroxide compound makes it necessary to use a common diluent in step (a) . . . Methanol is particularly preferred" (Strebelle, col. 2, ll. 54-65).

4. Strebelle teaches that the "peroxide compound which can be used in step (a) of the process according to the invention can be chosen from hydrogen peroxide and any peroxide compound containing active oxygen and capable of carrying out epoxidation. Examples which may be mentioned are the peroxide compounds obtained by oxidation of organic compounds such as ethylbenzene, isobutane and isopropanol" (Strebelle, col. 2, ll. 29-35).

5. Fukuda teaches:

> A method for the production of epichlorohydrin which is characterized in that, in a method for the production of epichlorohydrin from allyl chloride and an alkyl hydroperoxide in the presence of a catalyst, the allyl chloride is employed in the reaction after reducing the concentration of 1,5-hexadiene contained therein to 0.2 wt% or less.

(Fukuda Trans. 1, Claim 1).

6. Fukuda teaches that "commercially, a highly pure epichlorohydrin product is demanded and the elimination of impurities is essential" (Fukuda Trans. 3 ¶ 0004).

7. Fukuda teaches that "1,5-hexadiene is readily epoxidized in the reaction with the alkyl hydroperoxide and converted to 1,2-epoxy-5-hexene; which is difficult to separate from epichlorohydrin" (Fukuda Trans 4 ¶ 0008).

8. Fukuda teaches that "in a method for the production of epichlorohydrin from allyl chloride and an alkyl hydroperoxide in the presence of catalyst, the allyl chloride is employed in the reaction after first reducing the concentration of the 1,5-hexadiene contained therein to 0.1 wt% or less" (Fukuda Trans. 4 ¶ 0006).

9. Fukuda teaches that "in the reaction between the allyl chloride and the alkyl hydroperoxide in the presence of catalyst, the concentration of the 1,5-hexadiene contained in the allyl chloride is reduced beforehand to 0.1 wt% or less, and so it is possible to produce highly pure epichlorohydrin .by an ordinary distillation process" (Fukuda Trans. 4 ¶ 0007).

10. Fukuda teaches that "[w]hile it is most desirable that there essentially be no 1,5-hexadiene present in the distilled and purified allyl chloride, the concentration should be no more than 1,000 ppm and more preferably no more than 500 ppm" (Fukuda Trans. 5 ¶ 0009).

11. Fukuda teaches that the "alkyl hydroperoxide employed will be cumene hydroperoxide, ethylbenzene hydroperoxide, tert-butyl hydroperoxide, or cyclohexyl hydroperoxide and, optionally, the alkyl

hydroperoxide may be employed diluted with solvent" (Fukuda Trans. 5 ¶ 0010).

12. Table 1 of the Specification is reproduced below:

Table 1

	H_2O_2 Degree of conversion (%)	
Time (h)	Example 1 "standard" ALC	Example 2 "high purity" ALC
6	75.7	84.4
27	26.8	44.7
100		28.3
147		25.1

Table 1 discloses the degree of conversion of H_2O_2 at different times using "standard" allyl chloride and 180 ppm allyl chloride.

Principles of Law

The question of obviousness is resolved on the basis of underlying factual determinations including: (1) the scope and content of the prior art; (2) the level of ordinary skill in the art; (3) the differences between the claimed invention and the prior art; and (4) secondary considerations of nonobviousness, if any. *Graham v. John Deere Co.*, 383 U.S. 1, 17 (1966).

The Supreme Court has emphasized that "the [obviousness] analysis need not seek out precise teachings directed to the specific subject matter of the challenged claim, for a court can take account of the inferences and creative steps that a person of ordinary skill in the art would employ." *KSR Int'l v. Teleflex Inc.*, 550 U.S. 398, 418 (2007).

As noted by the Court in *KSR*, "[a] person of ordinary skill is also a person of ordinary creativity, not an automaton." 550 U.S. at 421.

Analysis

Prima facie obviousness

Strebelle teaches a method for manufacturing 1,2-epoxy-3-chloropropane (i.e., epichlorohydrin) by reacting allyl chloride and a peroxide, preferably hydrogen peroxide, in the presence of the zeolite catalyst and a solvent (FF 1-3). Strebelle teaches that other peroxide compounds which contain an active oxygen and result in epoxidation may be used (FF 4).

The Examiner acknowledges that Strebelle does not teach selection of allyl chloride with less than 2000 ppm by weight of 1,5-hexadiene (Final Rej. 4).

Fukuda teaches that 1,5-hexadiene is a contaminant found in allyl chloride (FF 9-10) and that "1,5-hexadiene is readily epoxidized in the reaction with the alkyl hydroperoxide and converted to 1,2-epoxy-5-hexene; which is difficult to separate from epichlorohydrin" (Fukuda Trans 4 ¶ 0008; FF 7). Fukuda recognizes this as a problem, noting that that "commercially, a highly pure epichlorohydrin product is demanded and the elimination of impurities is essential" (Fukuda Trans. 3 ¶ 0004; FF 6). Fukuda therefore teaches to reduce the 1,5-hexadiene levels to 500 ppm to avoid the epoxidation reaction with alkyl hydroperoxide (FF 7, 10).

In view of Strebelle and Fukuda, it would have been obvious to minimize the presence of a known contaminant in allyl chloride, 1,5-hexadiene, which would reasonably be understood to be readily epoxidized by peroxide compounds present in Strebelle's method and form impurities in

the final epichlorohydrin product which are difficult to remove (FF 1-4, 6, 7, 10).

Appellants argue that "in a three component reaction - reactant, oxidizing agent and catalyst - Strebelle and Takehisa differ significantly in their choice of two of the three. These are not analogous reactions. While the product of the two reactions is the same, the mechanisms of the reactions are necessarily different" (App. Br. 5). Appellants argue that "[t]his noncombinability of Strebelle and Takehisa is further evident in view of the 1998 article by Sheldon, originally submitted to the Examiner on October 29, 2008, wherein Takehisa's catalysts are described as ineffective with Strebelle's peroxide" (Reply Br. 5).

We are not persuaded. We recognize that Strebelle and Fukuda differ, but both Strebelle and Fukuda use allyl chloride (FF 1, 5) and both Strebelle and Fukuda incorporate peroxide compounds which contain active oxygen and are capable of carrying out epoxidation (FF 4, 7, 11). The ordinary artisan would reasonably have recognized that the 1,5-hexadiene contaminant in Fukuda's allyl chloride which was epoxidized by a peroxide compound into a contaminant would be equally susceptible to epoxidation by the peroxide compounds found in the method of Strebelle into contaminants in the final chlorohydrin product (FF 1-11).

The Sheldon[3] article does not address this fundamental point, but rather states that Fukuda's catalysts are ineffective with aqueous hydrogen

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[3] Sheldon et al., *Heterogeneous Catalysts for Liquid-Phase Oxidations: Philosphers' Stones or Trojan Horses*, 31 ACCOUNTS OF CHEMICAL RES. 485-493 (1998).

peroxide while TS-1, the catalyst used by Strebelle, is functional (see Sheldon 485, col. 2). Whether Fukuda's catalysts are effective with Strebelle's peroxide or not is unpersuasive on whether Fukuda suggested contaminant reduction. It is Fukuda's teaching that the presence of 1,5-hexadiene in allyl chloride will form a contaminant when reacted in the presence of a peroxide compound which renders obvious the removal of 1,5-hexadiene from Strebelle's reaction which includes a peroxide compound which can epoxidize 1,5-hexadiene into a contaminant (FF 1-11).

Unexpected Results

Appellants argue that "unexpected improvement in catalyst life as provided by the present invention has been amply demonstrated in the present specification" (App. Br. 6). Appellants argue that "production using normal allyl chloride is exhausted at 27 hours, while allyl chloride according to the present invention remains active at 147 hours of reaction time" (App. Br. 7).

The results in Table 1 of the Specification compare two examples, a "standard" allyl chloride with 2700 ppm 1,5-hexadiene and a "high purity" allyl chloride with 180 ppm 1,5-hexadiene (Spec. 7, ll. 10-28; FF 12). While the results may be compared with an allyl chloride that is reasonably understood as equivalent to that used by Strebelle, and therefore the closest prior art, the evidence of record does not demonstrate that the unexpected results are commensurate in scope with the degree of protection sought. *See In re Harris*, 409 F.3d 1339, 1344 (Fed. Cir. 2005) (Unexpected results must also be "commensurate in scope with the degree of protection sought by the claimed subject matter.")

As in *Harris*, where the unexpected results represented a single point along a range, the evidence of Table 1 of the Specification at best demonstrates that 180 ppm 1,5-hexadiene results in unexpectedly superior catalyst life, but provides no commensurate information about amounts up to 2,000 ppm, which is the value found in Claim 1.

Since we are persuaded that the results are unexpected for 180 ppm, we will reverse the rejection over Claim 3, which is limited to "wherein the allyl chloride comprises less than 200 ppm by weight of 1,5-hexadiene". However, we will affirm the rejection over Claim 1 because the unexpected result is not commensurate in scope with the limitation in Claim 1 "wherein the allyl chloride comprises less than 2000 ppm by weight of 1,5-hexadiene."

Conclusion of Law

(1) The evidence support the Examiner's conclusion that the cited references would have suggested the method of claim 1.

(2) Appellants have not presented evidence of unexpected results that outweighs the evidence supporting a conclusion of obviousness for Claim 1, but Appellants have presented evidence of unexpected results that outweighs the evidence supporting a conclusion of obviousness for Claim 3.

SUMMARY

In summary, we affirm the rejection of claim 1 under 35 U.S.C. § 103(a) as obvious over Strebelle and Fukuda. Pursuant to 37 C.F.R. § 41.37(c)(1)(vii)(2006), we also affirm the rejection of claims 2 and 4-16 as these claims were not argued separately.

We reverse the rejection of claim 3 under 35 U.S.C. § 103(a) as obvious over Strebelle and Fukuda.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a)(1)(iv)(2006).

AFFIRMED-IN-PART

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